

Project Final Report

FA2386-09-1-4067

Title: Multifunctional Composite Nanofibers for Smart Structures

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1. Background and Objectives

Functional materials are a new family of materials whose physical and chemical properties are sensitive to the change in the environment such as temperature, pressure, electric field, magnetic field, optical wavelength, adsorbed gas molecules and the pH value. The functional materials utilize the native properties and functions of their own to achieve an intelligent action. Functional materials in the form of fibers bring additional attribute as a carrier of the functions to higher order structures for applications ranging from energy harvesting and storage devices to flexible sensor network for vehicle and aircraft skin structures.

The overall aim of this study is to demonstrate the scalability and reproducibility of the Functional Composite Nanofiber (FCNF) concept using superparamagnetic nanofiber and carbon-nanotube nanofibers as examples. By collaborating with researchers in Japan and the US we further explore the applicability of the co-electrospinning process to other functional materials such as the Shape Memory Alloy (SMA) and/or shape memory polymer (SMP) and other hybrid systems. We specifically attempt to answer the following questions: (1) Can the composite nanofibers be electrospun with controllable diameter and morphology? (2) Can the properties of the nanoparticle be translated to the composite nanofibrous structures in the form of nonwovens and yarns? (3) Can these functional composite fibers be integrated into electronic devices? This proposed study is expected to lay the foundation for a more comprehensive understanding of the processing, structure and properties relationship of a large family of FCNF thus leading to more creative exploitation of the functions of FCNF for various smart structures.

Previous Related Work

With the support from AOARD in 2008, we successfully answered the first two questions and demonstrated the feasibility of fabrication and characterization of functional composite nanofibers containing superparamagnetic nanoparticles, silver nanoparticles and carbon nanotubes. We specifically demonstrated that electrospinning is an effective process to introduce new materials properties into ultrafine fiber form thus providing an effective means to carry and transfer nano-effects macro level performance.

Specifically composite nanofibers with embedded iron oxide nanoparticles were co-electrospun into nonwoven mat and over meter long yarn. The SEM and TEM image in

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14. ABSTRACT Encouraged by the successful fabrication and characterization of functional composite nanofibers we proceeded to further optimize the continuous yarn fabrication process and characterize the properties of the yarns from composite nanofibers. With the functional composite nanofiber structures (yarn and nonwoven) characterized we explored the feasibility of integrating these functional composite fibers into electronic devices and higher order structures. To facilitate the formation of complex device shapes the technology of converting composite nanofiber yarns to 3D braided structures was also explored. Specifically the objective of this two-year program was to develop the enabling composite nanofiber based materials for smart sensors/actuators and smart structures. This objective was achieved through (1) Fabrication and optimization of composite linear and 3D fibrous structures; (2). Exploration of the feasibility of nanofiber based super capacitor and Lithium Ion battery; (3) Characterization and validation of materials and sub element performance using EMI shield as an example; and (4) Development and integration of components for chemical sensor.					
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Fig 1 shows the uniform nanofiber and uniform distribution of iron oxide nanoparticles in carbonized PAN nanofibers.

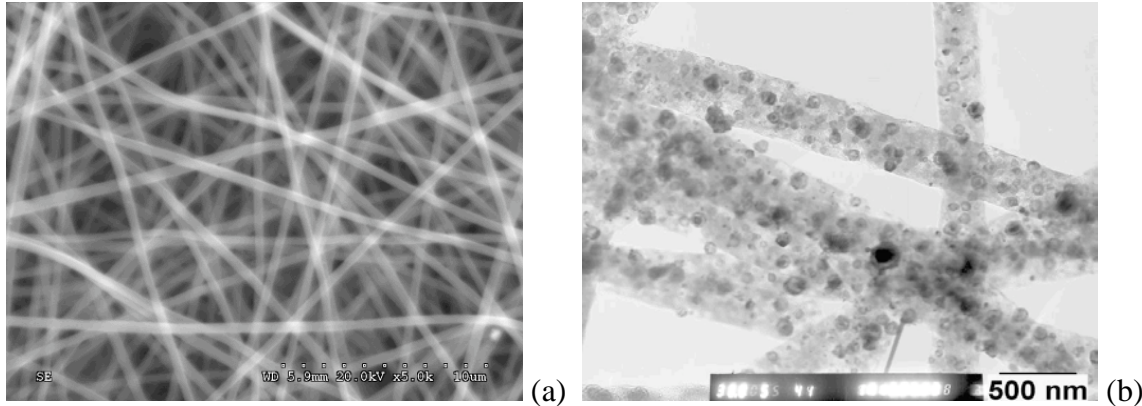


Figure 1. (a) SEM image of Carbonized Fe_3O_4 nanofibers; (b) TEM image of Carbonized Fe_3O_4 nanofibers

It can be seen in Fig 2 that the characteristic of highly sensitive superparamagnetic response to external magnetic field of the nanoparticless are manifested in the magnetic field response of the composite nanofibers. With incorporation of the carbon precursor, new highly electric conductive superparamagnetic composite nanofibers were fabricated. In situ synthesized silver nanoparticles and carbon nanotubes were also attempted to create conductive composite nanofibers.

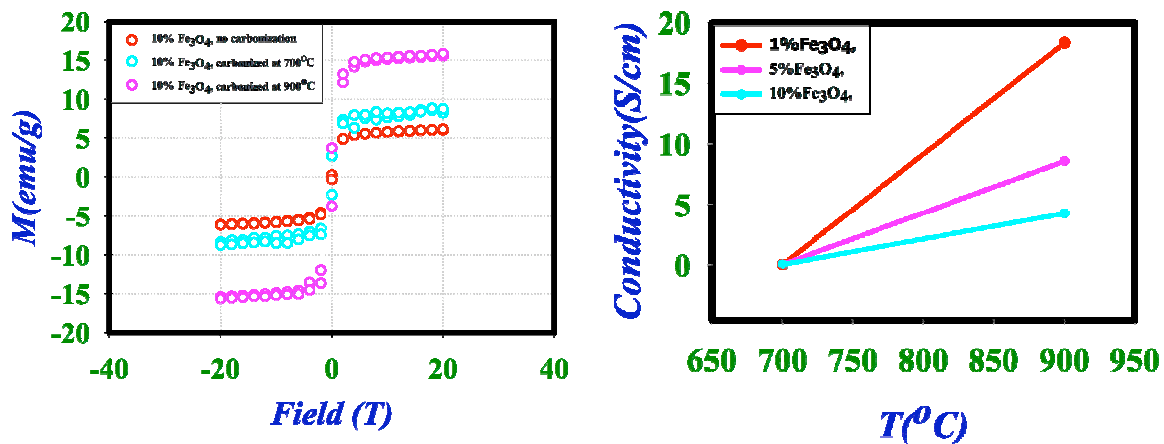


Figure 2.(a) Magnetic field response of Carbonized Fe_3O_4 nanofibers; (b) Electrical conductivity of Carbonized Fe_3O_4 nanofibers at various carbonization temperatures

2. Project Abstract

Nano-particles of different sizes, shapes and functions can be connected to macrostructures by co-electrospinning of the nanoparticle with polymers to form functional composite nanofiber without compromising the virgin nano-effect of the nanopartilcles. The successful demonstration of this phenomenon in professor Ko's laboratory has open up new opportunities of the engineering and manufacturing of a wide range of flexible smart structure capable of multiple functions such as sensing and

actuating for morphing structure.; EMI shielding; power generation, transmission and storage, as well as sensing/protection against chemical-biological and ballistic threats.

In order to facilitate the translation of the functional composite nanofiber concept from laboratory to production environment this proposed program is dedicated to demonstrate the scalability and reproducibility of functional composite nanofiber concept using magnetic nanofiber/electric conductive nanofiber as examples. Exploratory work were carried out on shape memory polymer composites. This study has laid the foundation for subsequent long term programs to develop a more comprehensive understanding of the processing, structure and properties relationship of a large family of functional composite nanofiber thus leading to more creative exploration of the functions of functional composite nanofiber for various applications.

3. Introduction

Encouraged by the successful fabrication and characterization of functional composite nanofibers, assemblies we proceed to further optimize the continuous yarn fabrication process and characterize the properties of the yarns from composite nanofibers. With the functional composite nanofiber structures (yarn and nonwoven) characterized we explored the feasibility of integrating these functional composite fibers into electronic devices and higher order structures. To facilitate the formation of complex device shapes the technology of converting composite nanofiber yarns to 3D braided structures was also explored.

Specifically the objective of this two-year program was to develop the enabling composite nanofiber based materials for smart sensors/actuators and smart structures. This objective was achieved through (1) Fabrication and optimization of composite linear and 3D fibrous structures; (2). Exploration of the feasibility of nanofiber based supercapacitor and Lithium Ion battery; (3) Characterization and validation of materials and subelement performance using EMI shield as an example; and (4) Development and integration of components for chemical sensor .

4. Nanofiber based device sub-element demonstration

4.1 Nanofiber based supercapacitor, Lithium Ion battery and transparent electrodes for solar cell (student – Nicole Lee; collaborator – professor John Madden)

Composite nanofibers are known for their high surface area to volume ratio, high porosity, and multi-functions, which are important features for enhanced surface reactivity and versatile applications. In this report, carbon nanofibers (CNF) composited with polypyrrole (Ppy) and silicon nanoparticles (SiNP) are applied in supercapacitor cathode and lithium ion battery (LIB) anode, respectively, to demonstrate the potential of nanofibers in the areas of electronics.

Supercapacitor

Supercapacitors, known for their high power densities, are promising candidate for compensating power deficiencies of batteries or fuel cells in hybrid vehicles. Reducing their cost and increasing energy density are major challenges for greater adoption of

supercapacitors. Among many materials, Ppy are chosen in our study for its high energy density, ease of fabrication, and low cost. It is coated onto the surface of CNF to use as a supercapacitor cathode. The high porosity and surface area of nanofiber composite facilitates ion diffusion and greater electrolyte insertion for higher energy storage. Pure Ppy film was also electrochemically deposited onto the surface of glassy carbon (GC) to compare the effect of Ppy morphology on charging/discharging speed. Both CNF/Ppy and GC/Ppy possess an intrinsic capacity of 150 – 180 F/g at slow cycle rate. However, the conductive CNF significantly improve the charging speed of Ppy at its reduced, less conductive state, as seen in the cyclic voltammetry curves of Figure 1. The fast rising of CV curve (dashed curve) from -0.8 – 0.6 V implies a less resistive nature of CNF/Ppy, which permits fast rising of charging current. Its CV also shows a similar trend at positive voltage end. Moreover, the time constant calculation further shows that nanofibrous Ppy reduces the ionic time constant of film Ppy by at least 100 \times .

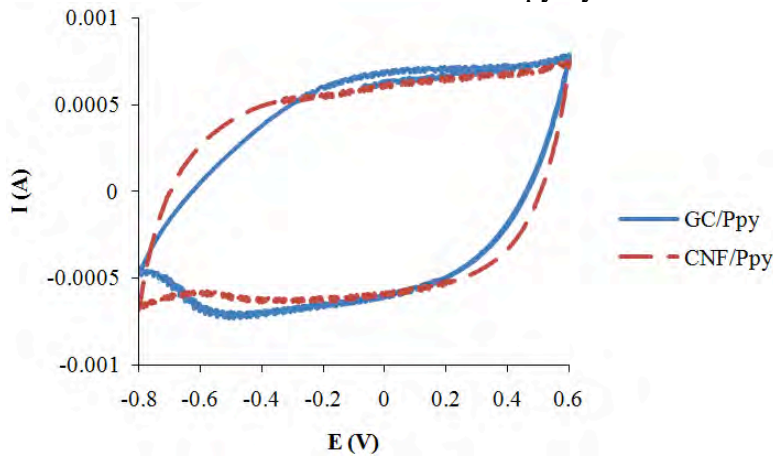


Figure 1 CV comparison of GC/Ppy and CNF/Ppy at 1 mV/s scan rate.

Lithium Ion Battery

LIB is a promising candidate to use as a long term energy source for electric vehicles due to its high energy density, low discharge rate, low maintenance issues, light weight, and small volume. However, in order to achieve the performance of conventional gasoline vehicles, it is necessary to develop LIB with much higher energy density. Silicon, known for its highest theoretical specific capacity of 4200 mAh/g comparing to other inorganic materials, is promising to replace graphite electrode, a standard anode in industry with 372 mAh/g theoretical capacity. In this study, silicon nanoparticle (SiNP) was used as the main energy storage material and was incorporated into carbon nanofibers through core-shell electrospinning. As shown in Figure 2(a), by protecting and separating SiNP with a thin layer of carbon shell, ca. 50 nm, SiNP fusion, pulverization, and capacity loss can be minimized during electrochemical cycling. Carbon is also ductile in absorbing the mechanical stress induced from SiNP expansion upon alloying with Li ions. The cycling performance of core-shell CNF/SiNP composite is compared with that of non-core-shell CNF/SiNP electrospun from single spinning dope. In general, core-shell CNF/SiNP shows improved capacity retention and stability. The result from cycling at 200 mA/g current density is shown in Figure 2(b). The core-shell sample retains ca. 200 mAh/g higher capacity than non-core-shell sample at the 20th cycle. Scanning electron

microscopy images of samples after cycling also show that core-shell CNF/SiNP retains more fibrous structure, while non-core-shell fibers fuse together through the agglomeration of surface SiNP.

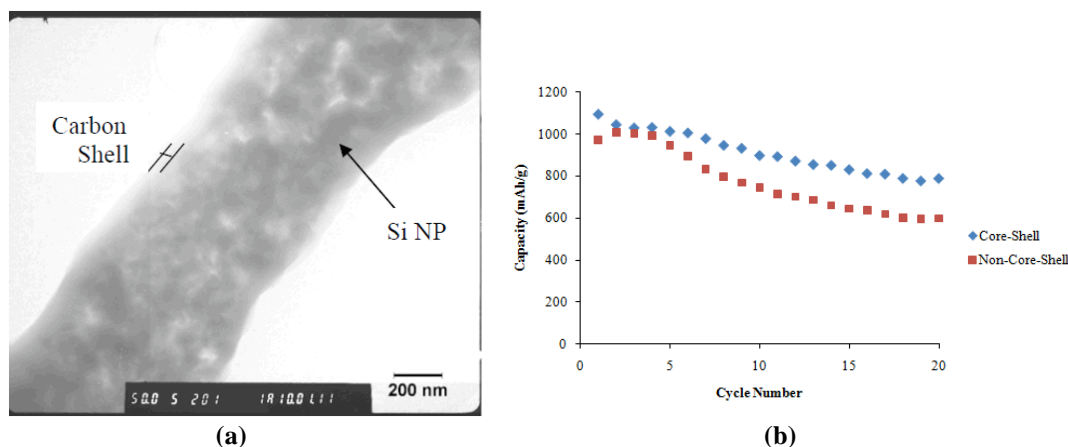


Figure 2 Core-shell CNF/Si composite with a thin layer of carbon shell coating on SiNP (a) and the capacity of both core-shell and non-core-shell CNF/SiNP electrochemically cycled at 200 mA/g rate (b).

Publications and Awards

1. Lee N., Fekri N., Fok E., Yang H., Madden J., Ko F. (2010). Polypyrrole coated carbon nanofibers for supercapacitor electrodes. paper presented at the Society for the Advancement of Material and Process Engineering (SAMPE), Seattle, WA. (2010)
1. Won the third place finalist in the SAMPE Graduate/Senior Student Award and Symposium Competition in the Master student category (2010)

4.2. Transparent Electrodes (students- Justin Richie and Joel Merten; collaborator- Peyan Servati)

Developing a durable and scalable transparent conductor (TC) with high optical transmission and low sheet resistance is a significant challenge for enabling next generation solar cell devices. High performance fibrous composite materials based on a carrier polymer with embedded functional nanostructures has the potential to serve as TC with high surface area, and can be deposited by the novel and scalable process of electrospinning. This work presents the development of a fibrous TC, where polyacrylonitrile (PAN) is used as a carrier polymer for multi-walled carbon nanotubes (MWCNT) to create electroactive nanofibers 200-500nm in diameter. Once carbonized, thin layers of this material has a low sheet resistance and high optical transmission. It is shown that in a two-stage carbonization process, the second stage temperature of above 700C is the primary factor in establishing a highly conductive material and single layers of nanofibers are typically destabilized at high temperatures. A high performance TC has been developed through optimizing carbonization rates and temperatures to allow for

single nanofiber layers fabricated by electrospinning MWCNT/PAN solutions onto quartz. These TCs have been optimized for concentrations of MWCNTs less than 20% volume fraction with above 90% transmissivity and sheet resistances on the range of several $\text{k}\Omega/\square$. The required MWCNT loading is well below that for TCs based on random networks of MWCNTs.

Publications

1. J. Ritchie, J. Mertens, H. Yang, P. Servati, F. Ko, Electrospun Composite Nanofiber Transparent Conductor Layer for Solar Cells, Presented at MRS, San Francisco, Spring 2011
2. J. Ritchie, J. Mertens, P. Servati, F. Ko Development of Nanofiber Composite Transparent Conductor for Photovoltaic Devices, E-MRS Spring and Bilateral 2011 Meeting

4.3. Electromagnetic fibers for EMI Shielding and Sensing (Student-Masoumeh bayat Collaborator-Dave Michelson)

Multifunctional composite nanofibers containing magnetite (Fe_3O_4) nanoparticles are developed in this work. The multifunctional composite nanofibers are proved to be electrical conductivity and magnetically permeable. Polyacrylonitrile (PAN) is used as an appropriate polymer which is capable of being pyrolyzed to produce electrically conductive carbon nanofiber matrix. In order to develop magnetic nanofibers, various amounts of Fe_3O_4 nanoparticles ranging from 3 to 10wt.% are embedded in the PAN nanofiber matrix. In addition, the electromagnetic behaviour of nanocomposites made of two different sizes (GA:20-30nm and GB:10-20nm) of Fe_3O_4 nanoparticles is examined. Electrospun composite nanofibers are thermally treated at both 700°C and 900°C to produce electromagnetic carbon nanofiber composites.

The composite nanofibers are characterized using scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffractometry (XRD), Raman spectroscopy, four-point probe and [Superconducting Quantum Interference](#) Device (SQUID). Electromagnetic Interference Shielding Effectiveness (EMI SE) of the pristine carbon nanofibers as well as electromagnetic composite nanofibers is examined using Vector Network Analyzer with TRL calibration.

Uniform nanofibers are obtained for all samples with choosing 10wt.% PAN concentration with larger fiber diameters for composite nanofibers as compared with pristine carbon nanofiber. The magnetic properties of two sizes of Fe_3O_4 nanoparticles are successfully transferred into the as-spun Fe_3O_4 /PAN composite nanofibrous structure. The electromagnetic properties of the composite materials are tuned by adjusting the amount and size of Fe_3O_4 nanoparticles in the matrix and carbonization process. XRD and Raman results revealed that graphitization of carbon matrix can be enhanced by the presence of iron oxide and by increasing the pyrolysis temperature. However, very minor changes of graphitization enhancement are acquired for composites fabricated with smaller Fe_3O_4 nanoparticles (10-20nm). By embedding 10wt.% of 20-30nm Fe_3O_4 nanoparticle, saturation magnetization of 16emu/g is obtained with electrical conductivity of 9.2S/cm for carbonized composite nanofiber at 900°C. However, it decreases to 9.0emu/g and 1.96S/cm for composite made of 10wt.% 10-20nm Fe_3O_4 nanoparticle

carbonized at 900°C. The high surface area provided by the ultrafine fibrous structures, the flexibility and tuneable electromagnetic properties are expected to enable the expansion of the design options for a wide range of electronic devices such as sensors and actuators as well as Electromagnetic Interference Shielding Effectiveness (EMI SE). The electromagnetic composite nanofibers are demonstrated to act as strong electromagnetic interference shield of up to 70-80dB. The main shielding mechanism is found to be absorption of electromagnetic wave rather than reflection. The shielding also enhances with Fe₃O₄ content in the carbon nanofiber matrix.

Publications

1. M. Bayat, H. Yang, E. Folk, N. Lee, F. Ko, “Electrospinning of Iron Oxide Nanofibre Composites”, Poster presentation, Fibre Society Conference 2008, Montreal, Canada, October 1st -3rd, 2008.
2. M. Bayat, H. Yang, F. Ko, “Electromagnetic Fe₃O₄/Carbon Nanofibre Composites”, Invited Student Paper Presentation, Pacific Centre for Advanced Materials and Microstructures (PCAMM 2009), SFU, Canada, December 12th, 2009.
3. M. Bayat, H. Yang, F. Ko, “Structures and properties of Fe₃O₄-carbon multifunctional composites nanofibers”, ICCM 2009.
4. M. Bayat, H. Yang, F. Ko, “Electrical and Magnetic Properties of Carbon/Fe₃O₄ Nanofibre Composites”, December 2009.
5. M. Bayat, Heejae Yang, Frank Ko, “Electrical and Magnetic Properties of Carbon/Fe₃O₄ Composite Nanofibres”, Poster presentation, The Minerals, Metals & Materials Society, TMS, February 2010.
6. M. Bayat, N. Lee, J. Mertens, J. Ritchie, H. Yang, F. Ko, P. Servati, “Nanofibers for Electricity Generation, Storage and Sensing”, poster presentation, Nanomaterials for alternative energy applications workshop, June 20, 2010.
7. M. Bayat, H. Yang, F. Ko, “Structure and properties of superparamagnetic composite nanofiber”, 8th Joint Canada-Japan Workshop on Composites, 2010.
8. F. Ko, M. Bayat, H. Yang, Multifunctional Composite Nanofibers”, Zhejiang, 2010.
9. M. Bayat, H. Yang, F. Ko, “Electrical and Magnetic Properties of Fe₃O₄/C Nanofibre Composites”, SAMPE 2010 Conference Proceedings: New Materials for a New Economy, Seattle, WA, May 17-20, 2010, Society for the Advancement of Material and Process Engineering.
10. F. Ko, M. Bayat, “Multifunctional Composite Nanofibers by electrospinning”, Proceedings of the ASME 2010 International Mechanical Engineering Congress & Exposition, IMECE2010, November 12-18, 2010, Vancouver, British Columbia, Canada.
11. M. Bayat, H. Yang, F. Ko, “Electromagnetic Properties of Electrospun Fe₃O₄/Carbon Composite Nanofibres”, [Volume 52, Issue 7](#), 23 March 2011, Pages 1645-1653, Polymer, 2011.

4.4. Nanofiber based chemical sensors (student-Jonas Flueckiger; Collaborator-Professor Karen Cheung)

This research program focuses on the development of a novel chemical sensor platform using nanofibre technology to increase sensitivity and reduce detection time compared to conventional detection methods. Nanofibres and their composites can

provide fundamental building blocks for the construction of devices and structures that perform unique new functions. This research focused on two aspects; the fabrication of nanofiber based sensing layers and on the integration with micro-electromechanical systems (MEMS) to provide the electrical interconnects to the new nanomaterials. The goal is to design robust manufacturing methods to make sure that large-scale production of the integrated sensor is possible.

The sensitivity and time response of the sensing film is proportional to the surface area of the film per unit mass. Nanofiber based sensing films have a one to two orders of magnitude larger sensing area per unit mass than solid films and can therefore offer faster response times and increased sensitivity. We fabricated nanofiber based films by electrostatic fabrication or electrospinning. It is a relatively simple and low cost way of fabricating long, continuous organic or inorganic nanofibers from a polymer solution. In this way, we processed to different metal oxides (TiO_2 , and ZnO) and a conductive polymer (polyaniline, PANi / polyethylene, PEO) into nanofibers with different morphologies (depending on the fabrication process parameters). The nanofibers have been characterized by SEM, TEM, and XRD. Furthermore we are currently investigating the electrical and sensing properties (sensitivity, selectivity, and response time). To do so the nanofibers are deposited on microfabricated interdigitated electrodes (IDE). The Au electrodes were fabricated through standard cleanroom processes: a) Patterning of the glass or silicone substrate with a photoresist (Shipley 1813) by photolithography, b) evaporation of gold with an additional chromium layer for better adhesion (Cr thickness 20 nm, Au thickness 150 nm), c) lift off and removal of photoresist in an acetone bath. In parallel a gas test chamber with appropriate environmental control (temperature, humidity, and gas concentration) has been designed and integrated with the electrical characterization equipment. We are now able to expose the sensor to different gases and gas mixtures at different concentrations and flow rates. DC measurements and impedance spectroscopy are both used to understand the detection mechanisms and to determine the sensitivity and response times of the nanofiber based sensing mats.

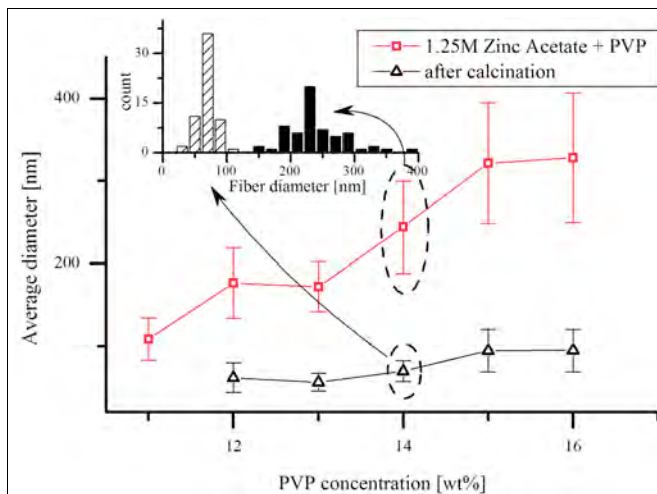


Fig 1.: Diameter distribution of ZnO nanofibers as function

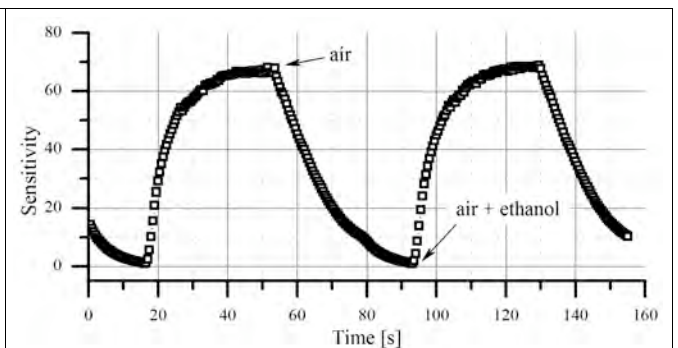


Fig 2.: Dynamic response of TiO_2 nanofibers exposed to air

of polymer solution (PVP in ethanol) concentration: The histogram is given for a 1.25 M Zinc Acetate / 14 wt% PVP solution

and air/ethanol.

Publications

1. J. Flueckiger, F.K. Ko, K.C. Cheung, Microfabricated Formaldehyde Gas Sensors, *Sensors*, 2009, 9(11), 9196-9215
2. J. Flueckiger, F.K. Ko, K.C. Cheung, Electrospun Electroactive Polymer and MetalOxide Nanofibres for Chemical Sensor Applications, ASME International Mechanical Engineering Congress and Exposition, 2010 (7 pages)
3. J. Flueckiger, F.K. Ko, K.C. Cheung, Electrospun Nanofibre Based Chemical Sensor: Electrical Characterization, The 33rd Conference of the Canadian Medical and Biological Engineering Society, June 15-18, 2010 (poster presentation)
4. J. Flueckiger, K.C. Cheung, F.K. Ko, Multifunctional Composite Nanofibres, 2010 CMOS Emerging Technologies Workshop, May 19-21, 2010, Whistler (invited talk)

4.5 Shape Memory Polymer Composite Structures (postdoc- Cagri Ayranci and Heejae Yang)

This task has two major components, namely: characterization of properties of polymers and their fiber composites with shape memory effect capabilities. The original plan was to collaborate with colleagues from Japan using their SMP. However the SMP was not forth coming. In the interest of time we used the shape memory polymer yarns provided by Professor Jinlian Hu of the Institute of Textiles and Clothing at the Hong Kong Polytechnic University. The yarns were composed of three SMP fibers each of which with approximately 40 micrometer diameters. The average longitudinal elastic modulus of the yarns was found to be 1.134 GPa (± 0.012 Standard Deviation) at room temperature. The low modulus nature of these SMP yarns calls for a material system that has higher stiffness values while still able to show shape memory effects to successfully build the MAV reinforcements.

We then started to investigate the feasibility of reinforcing polymers with shape memory effect using conventional advanced fibers such as Carbon and Kevlar®. Initial in-house experiments were conducted. Figure 3 shows the successful shape recovery during these proof-of-concept experiments conducted at the AFML.

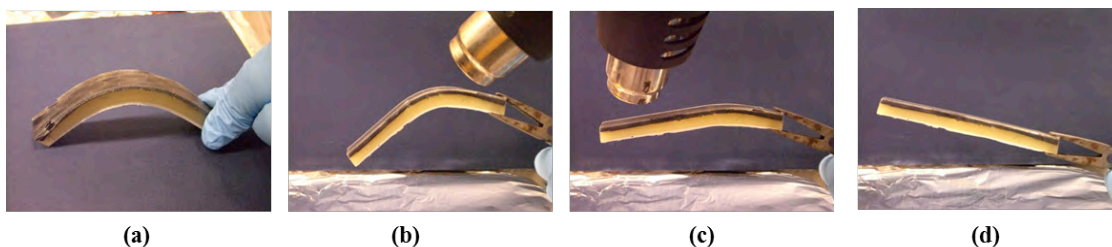


Figure 3: Demonstration of the shape recovery of SMPC composite beam. (a) Temporary curved shape; (b) and (c) application of heat and gradual shape recovery; (d) complete shape recovery and restructure of the permanent shape.

Later, a special mold was designed and built to produce a tubular braided structure, with a constant cross section throughout the length. This was an initial step for the tapered cross section tubular MAV reinforcements that will be built. A temporary curved shape was assigned to tube (Figure 3(a)) , and subsequently heat was applied (Figure 3 (a) and (b)). As a result, the shape recovery of the tubular braided composites were successfully achieved.

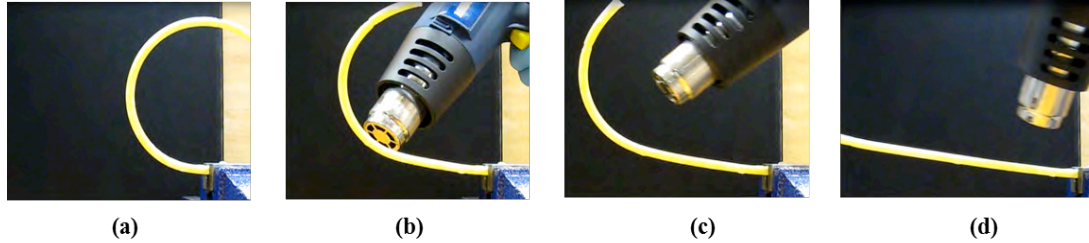


Figure 4: Demonstration of the shape recovery of tubular braided composite. (a) Temporary curved shape; (b) and (c) application of heat and gradual shape recovery; (d) shape recovery of the permanent shape (i.e. deployed shape of the MAV reinforcement).

Finally the preforms of the tubular braided reinforcements that are 250 mm in length and have tapered tubular cross sections (tapered from 4 mm down to 2 mm cross sectional diameter) have been prepared using a hexagonal braider at the AFML at UBC. They were impregnated using resin to form the reinforced composite tube. Figure 4 shows a zoomed-in picture of the said tapered structure.



Figure 5: Zoomed in picture of the tapered cross section braided reinforcement preform.

Glass transition temperature (T_g) is one of the most crucial properties that to be determined for applications of materials with shape memory effect. During this study, the effect of hardener content of the epoxy system on the glass transition temperature was investigated by changing the resin-to-hardener content of the epoxy system. Figure 5 shows the strong dependency of T_g on the degree of crosslinking of the polymer. This finding is crucial to demonstrate the tailorability of the resin for different applications. It is also of importance to understand the effect of reinforcement orientation on the glass transition temperature and shape recovery force of these composites. Hence, effect of fiber angle placement on glass transition temperature was investigated and findings have been submitted for publication [1]. The shift in the peaks of Tan Delta plots of Figure 6 is an illustration of this effect [1].

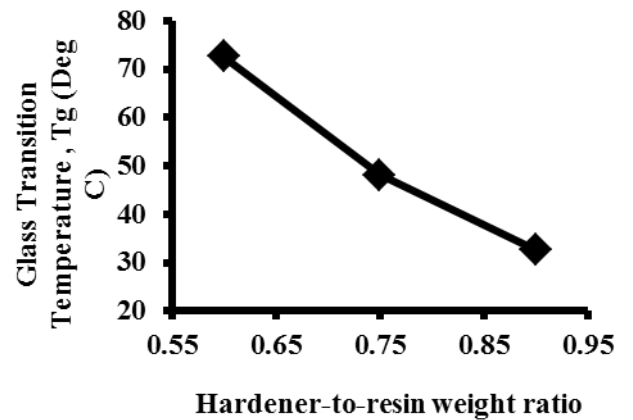


Figure 5: T_g versus hardener-to-resin weight ratio.

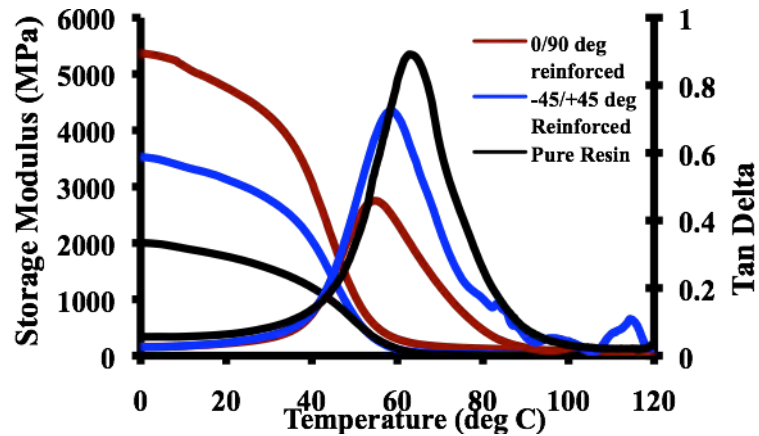


Figure 6: Storage modulus and Tan Delta versus temperature graphs obtained using Dynamic Mechanical Analyzer for pure resin and two different angle fiber placements on composites with shape memory effects.

Depending on the type of the application, it is often necessary to understand and characterize the shape recovery stress of the materials for design. The open literature on this topic is very limited; therefore, a set of controlled experiments has been designed. Findings will provide the necessary information to develop a model that predicts the recovery stress of these materials.

Activation methodologies of these materials are also a concern preventing broader use of these advanced materials; therefore, it is also important to investigate the self-activation capabilities of these materials. We have initiated a study regarding the use of reinforcing materials (e.g. Carbon fibers) for activating the shape memory effect where a current is passed through the reinforcements, rather than use of an external heat source that is not always readily available in possible applications sites of these materials. Initial findings have been submitted for publication [2].

Publications (accepted)

[1] Cagri Ayranci, Frank Ko, Tucker Howie, MinoruTaya, "Shape memory effect of a thermoset polymer and its fiber reinforced composites, submitted to the 18th International Conference on Composite Materials (ICCM 2011) that will be held in August 2011.

[2] Cagri Ayranci, Heejae Yang, and Frank Ko, "Self-activated Carbon Fiber Reinforced Shape Memory Polymer Composites", submitted to the 26th ASC Technical Conference (the Second Joint US-Canada Conference on Composites) that will be held in September 2011.

5. Acknowledgements

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